A STUDY OF THE SYNTHESIS OF THE ZEOLITIC IMIDAZOLATE FRAMEWORK MEMBRANE ZIF-11

A Senior Scholars Thesis

by

JOHN THORKELSON

Submitted to the Office of Undergraduate Research
Texas A&M University
in partial fulfillment of the requirements for the designation as

UNDERGRADUATE RESEARCH SCHOLAR

July 2011

Major: Chemical Engineering

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Approved by:

Research Advisor:	Hae-Kwon Jeong
Executive Director for Honors and Undergraduate Research:	Sumana Datta

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ABSTRACT

A Study of the Synthesis of the Zeolitic Imidazolate Framework Membrane ZIF-11.
(July 2011)

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With increasing energy consumption and fossil fuels being the primary energy source, there is need to invest in alternative sources of energy. Hydrogen gas (H_2) is a promising possibility because it is inexhaustible, efficient, low cost, and non-polluting. One of the greatest expenses in the production of hydrogen gas is in the separation and purification process to rid H_2 of any contaminants. Because of this, there is an increased interest in developing a cost-effective method to purify hydrogen. Zeolitic Imidazolate Framework (ZIF) membranes offer great potential for energy efficient and highly effective gas separation processes. ZIFs are microporous crystal structures, belonging to the family of metal organic frameworks (MOFs), that are highly chemically and thermally stable, and have uniformly distributed and sized pores, making them ideal candidates for gas separation. Demonstration of a general method for ZIF membrane synthesis is desired. ZIF-11 membranes are produced by growing ZIF-11 crystals, synthesized by mixing zinc nitrate hexahydrate and benzimidazole in a diethylformamide solution, on α-

alumina (Al_2O_3) supports. The process takes between 4 and 8 hours at a temperature of 100 °C. ZIF-11 crystals were synthesized in powder form and characterized using X-Ray diffraction (XRD). Membranes were synthesized, examined for defects using an optical microscope, and characterized by X-Ray diffraction. The results suggest that a general process for ZIF membrane synthesis has been developed, though a recipe for consistent ZIF-11 synthesis needs to be developed. Further experiments should be done to create large well-intergrown crystals on the membrane, and hydrogen gas permeation testing of ZIF-11 membranes should be investigated for eventual industrial applications.

DEDICATION

To my father, J.M. Thorkelson, my mother, R.C. Thorkelson, my grandmother, G.E. Carazzo, and to Meri Pashilk, the science teacher who made a difference for me. Each of these persons inspired and encouraged me to delve into the sciences and never stop asking the question “Why?”

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NOMENCLATURE

α-Al_2O_3	α-alumina
bIm	benzimidazole
DEF	diethylformamide
DMF	dimethylformamide
MOF	metal organic framework
MeOH	methanol
mIm	2-methylmidazole
NaCOOH	sodium formate
P	pressure
T	temperature
t	time
XRD	X-Ray diffraction
ZIF	zeolitic imidazolate framework
$Zn(NO_3)_2 \cdot 6H_2O$	zinc nitrate hexahydrate

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CHAPTER I

INTRODUCTION

As global energy consumption has increased, there has been great interest in pursuing alternative energy sources to fossil fuels. The global energy consumption is predicted to double by the year 2050, and with fossil fuels being a primary energy source, great political, environmental, and economic pressures have ensued [1]. Hydrogen gas (H_2) stores chemical energy in the H-H bond, and this energy is released when it combines with oxygen, forming water [2]. Hydrogen gas is a clean, efficient, inexhaustible and cost-effective energy carrier and has great potential both to become a long term energy solution and to help aid the economy in becoming less dependent on oil [2-4]. It should be noted that hydrogen is an energy carrier, not an energy source, similar to how electricity is a carrier of electrons, not a source of them. There are approximately 41 million metric tons of H_2 produced industrially each year, which constitutes almost 2% of the world's total energy [1]. Hydrogen gas is widely used in the refining industry for chemical production, desulfurization, and hydro-treating [5]. Large capital investment must be made in the production of H_2 in order to pay for separation and purification processes, thereby greatly increasing the cost of H_2.

This thesis follows the style of Journal of Membrane Science.

Membrane separation technology is a promising method for gas separation and has great potential to provide a method to separate and purify H_2 [1, 6-9].

Zeolitic Imidazolate Frameworks (ZIFs), a sub-class of Metal Organic Frameworks, are three-dimensional crystals made from metal ions, such as $Zinc^{2+}$ and $Cobalt^{2+}$, linked to Imidazolate to create tetrahedral frameworks [10-12]. The preferred Si-O-Si bond angle in Zeolites measures to be 145°, and coincidentally, the M-Im-M bond in ZIFs also measures to be approximately 145°, making it possible for ZIFs to have Zeolite-based topologies [13, 14]. Because the Imidazolate linkers are carbon based, it is also possible to introduce functionality to frameworks, making the study of ZIFs exciting and worthwhile. There are many Imidazolate-based linkers that can be used to synthesize ZIFs, which creates a vast number of possible ZIF structures [10]. Further complexity can be achieved by introducing multiple linkers in various concentrations into the solvent [15]. ZIFs have been found to be remarkably stable both chemically and thermally [16]. The range of pore size, number of possible structures, overall stability, design-ability, and potential for functionalization make ZIFs ideal candidates for gas separation. To date, very few ZIFs have been made into membranes and tested for gas separation properties, leaving an expansive number of possible membranes unexplored [17, 18]. One of the biggest obstacles in the synthesis of ZIF membranes is creating continuous crystal structure with minimal defects that create strong bonds with the support [19].

ZIF-11 is a Zeolitic Imidazolate Framework made from benzimidazole. It takes on the RHO Zeolite topology because of the steric hindrance induced by the benzyl group on the imidazole linker, and has a pore size of 3Å [10]. This pore size makes it an ideal candidate for H_2 gas separation, as the kinetic diameter of hydrogen gas is 2.89Å [20]. Furthermore, studies have demonstrated that ZIF-11 crystals exhibit reversible hydrogen sorption [21]. This study focused on the synthesis and characterization of ZIF-11 crystals, and the synthesis of the crystals on a membrane to provide further evidence for a general ZIF synthesis method, and for the purpose of eventually measuring gas separation properties [22].

CHAPTER II

METHODS

Required chemicals

Zinc nitrate hexahydrate ($Zn(NO_3)_2 \cdot 6H_2O$, >98%, Sigma-Aldrich) was used as a source of zinc. Benzimidazole ($C_7H_6N_2$, bIm, > 99%, Alfa-Aesar) was the primary ligand used. Sodium formate (NaCOOH, >95%, Sigma-Aldrich) was used as a deprotonator. Diethylformamide (DEF, 99%, TCI America) and Methanol (CH_3OH, >95%, Fischer Scientific) were used as a reaction solvent and sonication solvent respectively.

Synthesis and characterization of ZIF-11 powder

The recipe for the synthesis of ZIF-11 powder is a modification of the process developed by the Yaghi group, which does not use NaCOOH [13]. A solid mixture of 0.356 g of $Zn(NO_3)_2 \cdot 6H_2O$, 2.119 g of benzimidazole, and 0.122 g of NaCOOH were dissolved completely in 60 mL of DEF (molar ratio of 1:15:1.5:900) and placed in an oven at 100°C for four hours. The oven was then allowed to cool for two hours, after which the solution was removed, centrifuged, and carefully decanted, leaving the powder behind. The crystals were allowed to dry in an oven over night at 100°C. The powder was characterized by using a Rigaku Miniflex II powder X-Ray diffractometer (XRD) with Cu Kα radiation (λ=1.5406 Å).

Preparation of α-Alumina supports

α-alumina supports (also referred to as substrate disks) were made by putting 1.5 g of α-alumina powder under 7 tons of pressure in a hydraulic press for 1 minute. The disks were then placed in an oven and heated from 30°C to 100°C at a rate of 1°C/min. The disks were maintained at 100°C for 2 hours. The oven temperature was then increased to 400°C over a period of 3 hours and maintained at temperature for 2 hours. Finally, the oven temperature was increased to 700°C over 3 hours, maintained at temperature for 4 hours, and then allowed to cool down to room temperature.

Substrate modification process

Polished α-alumina substrate disks were placed in an oven at 200°C for two hours to dry. While the disks were still in the oven, a solution of 3.62 g of benzimidazole (or methylimidazole) in 50 mL of methanol was placed drop wise onto the polished side of the disk, wetting it completely. The oven door was closed and the supports were allowed to dry for approximately 20-30 minutes. This process was repeated between six and nine times, turning the α-alumina disks a light or dark brown color. The supports were sonicated in methanol after every three repetitions of dropping the methanol solution onto the disk to remove any excess ligands not bonded to the support [22]. Images of modified substrate disks are shown below in **Fig. 1.**

Fig. 1. α-alumina supports after surface modification. The left support was modified with a mIm solution and the right support was modified with a bIm solution.

Synthesis activation and characterization of ZIF-11 membranes

A solid mixture of 0.356 g of $Zn(NO_3)_2 \cdot 6H_2O$, 2.119 g of benzimidazole, and 0.122 g of NaCOOH (molar ratio of 1:15:1.5) were dissolved completely in 60 mL of DEF. A substrate disk was loaded vertically into a Teflon holder, placed in the membrane growth apparatus (shown below in **Fig. 2.**), immersed in the DEF solution, and placed in an oven at 100°C for four hours. The oven was then allowed to cool for two hours, after which the membranes were removed. To activate the membranes, the remaining solvent, DEF in this case has to be removed. This was done by submerging the membrane in pure methanol for 24 hours to allow for a solvent exchange of MeOH with DEF. Once the membrane was removed, the methanol evaporated, leaving the pores of the ZIF unobstructed. The membrane was then examined for cracks and defects with an optical microscope, and then characterized by using a Rigaku Miniflex II powder X-Ray diffractometer with Cu Kα radiation (λ=1.5406 Å).

Fig. 2. ZIF membrane synthesis autoclave apparatus. The white cylinders are the Teflon containers that contain the membrane support, the synthesis solution, and are encased by the metal pieces.

CHAPTER III

RESULTS

Characterization of ZIF-11 powder

ZIF-11 crystals were synthesized with the above recipe (molar ratio of 1:15:1.5) in three solvents, DEF, DMF, and MeOH. The resulting crystals were centrifuged, decanted, dried overnight, and characterized by X-ray diffraction (XRD) by comparing the measured pattern with reported data. The XRD patterns of the powder from the three solutions and the powder from the DEF solution measured from 3-20° are shown below in **Figs. 3** and **4** respectively. Only the DEF recipe produced the characteristic peaks indicative of ZIF-11 (peak below 5 degrees). The presence of this peak and others confirmed the desired crystals had been synthesized (peak locations are shown in **Table 1.**) [6].

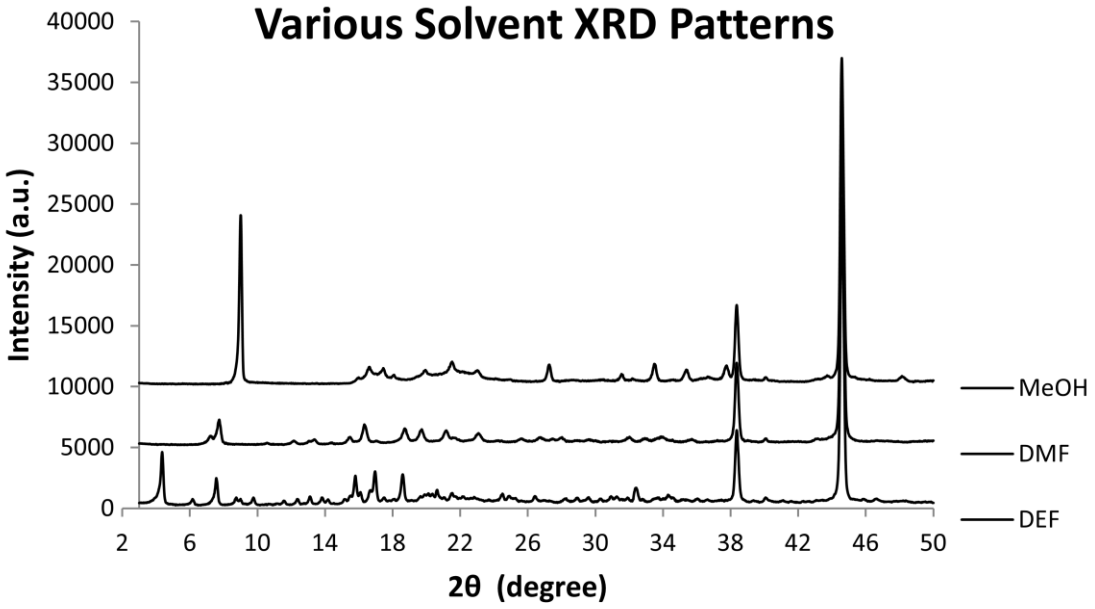


Fig. 3. X-Ray diffraction pattern from 3-50 2θ degrees of ZIF-11 powder grown in DEF, DMF and MeOH.

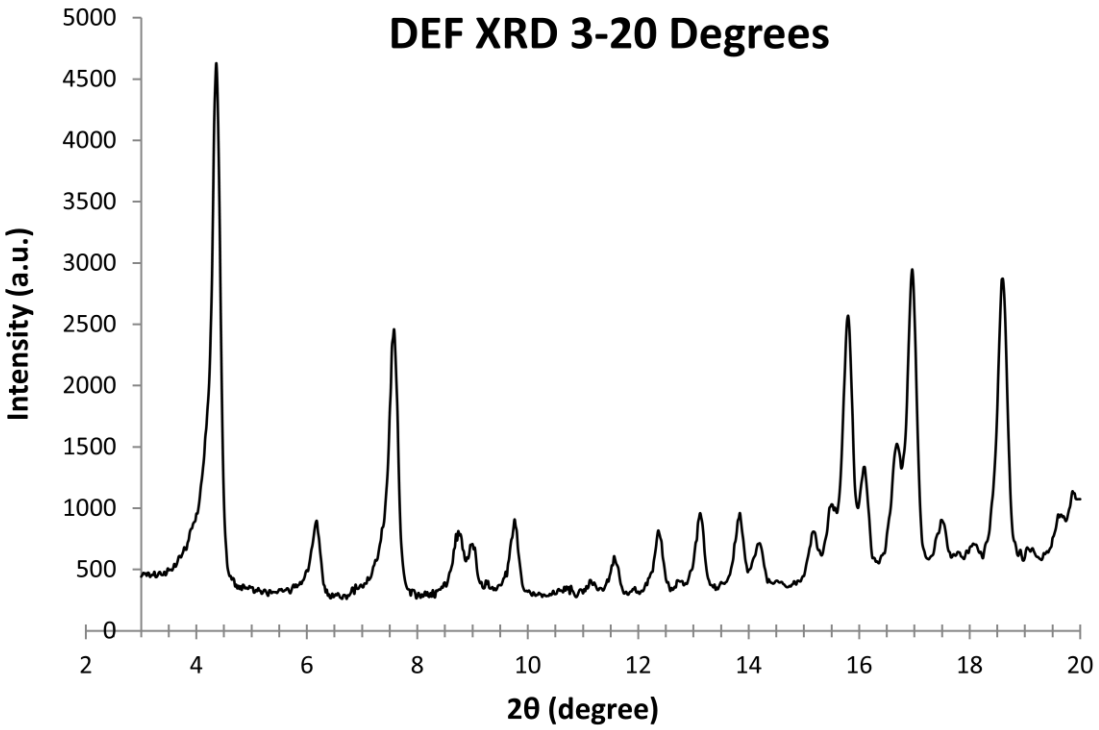


Fig. 4. X-Ray diffraction pattern of ZIF-11 powder grown in DEF from 3-20 2θ degrees.

Table 1. Observed and simulated peaks of ZIF-11 powder. Simulated peaks taken from supplemental information in Park et al [13].

Observed XRD Peaks	Simulated XRD Peaks
2θ	2θ
4.36	4.34
6.16	6.14
7.58	7.52
8.7	8.69
9.76	9.72
11.06	11.08
12.36	12.3
12.66	12.68
13.1	13.05
13.82	13.76
15.78	15.7
17.48	17.42
18.58	18.5

ZIF-11 membrane growth

The first attempt to synthesize a ZIF-11 membrane resulted in too few crystals on the support to get conclusive XRD patterns for characterization.

ZIF-11 membrane growth with doubled concentrations

In attempt to increase the number of crystals that could grow in the solution, the concentrations of the $Zn(NO_3)_2$, bIm, and NaCOOH from the original recipe were doubled. This experiment was also designed to observe the difference a mIm and bIm-modified substrate has of crystal growth. The XRD patterns (shown below in **Fig. 5.**) measured did not match the ZIF-11 pattern from the powder synthesized earlier or the literature. The mIm-modified membrane was very dark (see **Fig. 1.**). The powder collected from the bottom of the autoclave apparatus was characterized and had some of the desired ZIF-11 peaks. Optical microscopy images are shown below in **Fig. 6.**

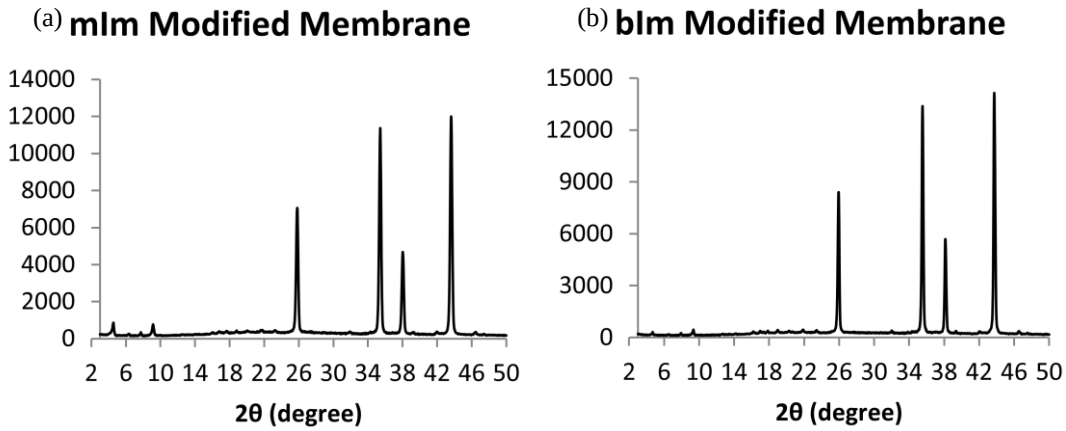
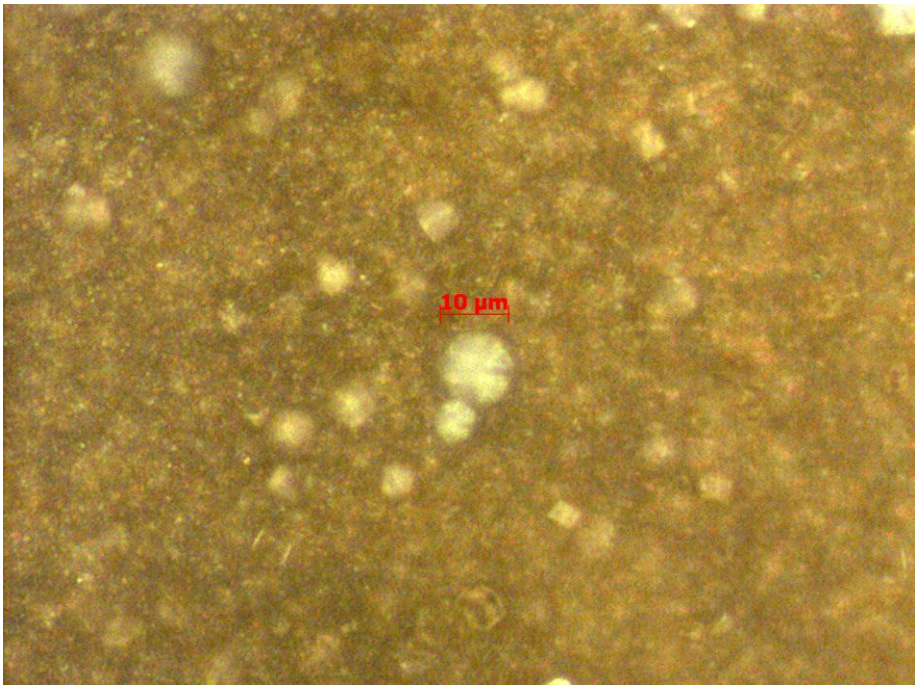


Fig. 5. X-Ray diffraction pattern of (a) mIm-modified ZIF-11 membrane and (b) bIm-modified ZIF-11 membrane. The characteristic peaks are not present indicating a ZIF-11 phase is not present.

(a)



(b)

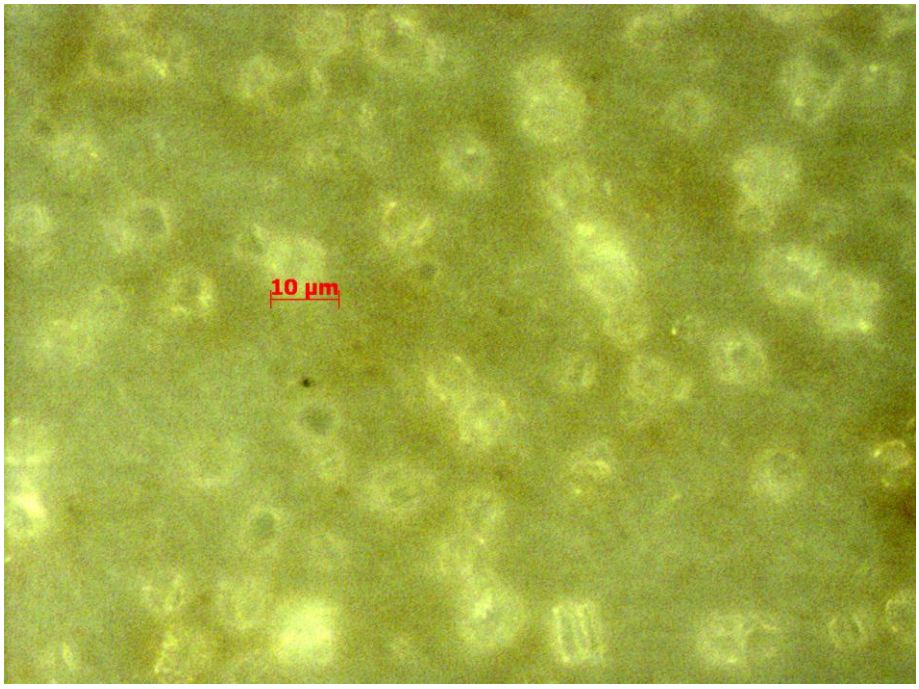


Fig. 6. Optical microscope images of (a) undesired crystal product on a mIm-modified support and (b) undesired crystal product on a bIm-modified support.

Effect of time on ZIF-11 membrane growth

Keeping to the original recipe and being sure not to make the supports too dark from to many applications of the mIm and bIm in MeOH solution, the synthesis time was adjusted to observe how the number of crystals is changed by reaction time. Six membranes were synthesized, three with mIm and three with bIm modification, at 8 hrs, 12 hrs, and 24 hrs reaction time. XRD measurements (shown below in **Figs. 7** and **8**) were taken and indicated that ZIF-11 was not formed.

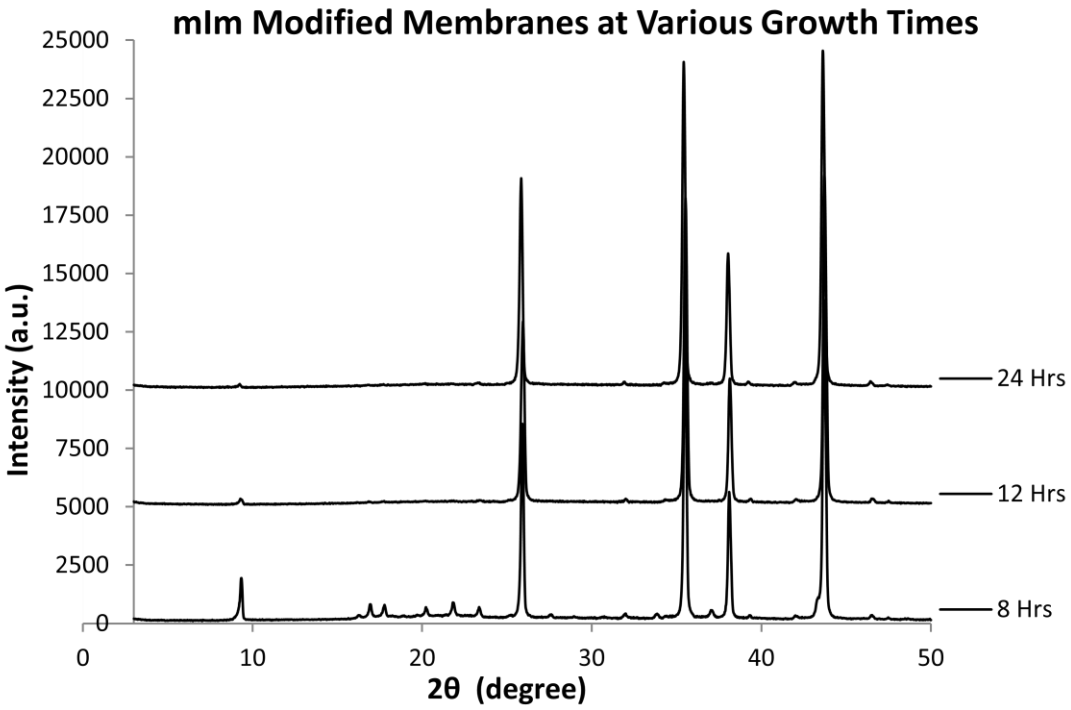


Fig. 7. X-Ray diffraction patterns of mIm-modified membranes grown for 8, 12, and 24 hours growth time.

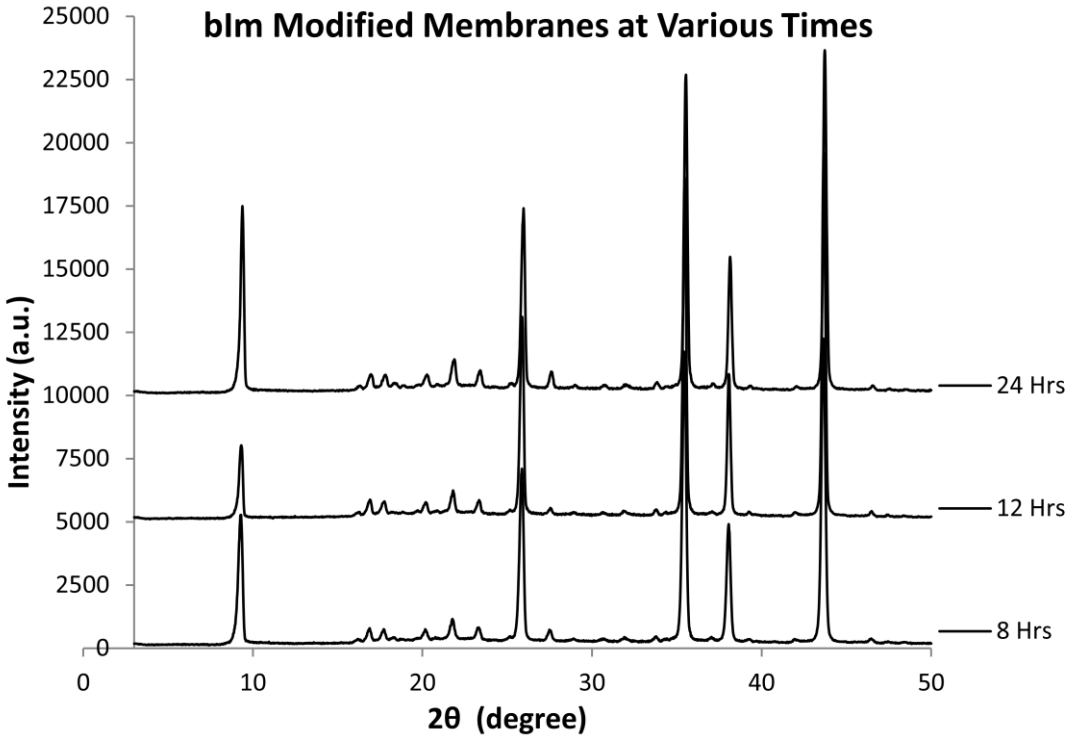
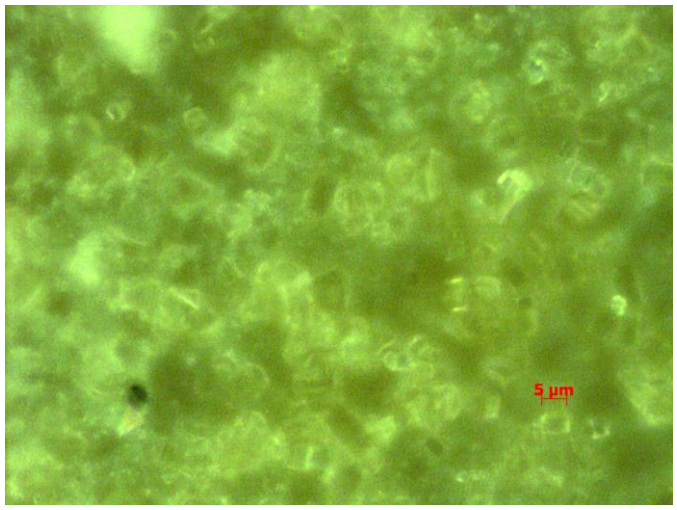


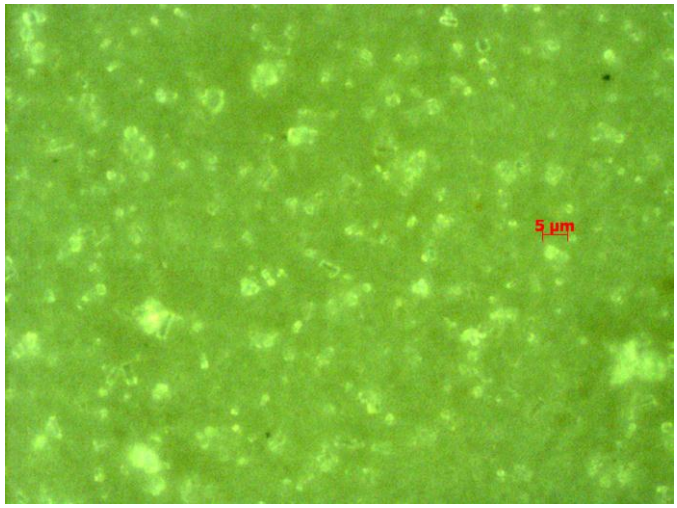
Fig. 8. X-Ray diffraction patterns of bIm-modified membranes grown for 8, 12, and 24 hours growth time.

The crystals on the mIm-modified membrane were not large and had large amounts of space in between them. Images of the mIm-modified membranes at various growth times are below in **Fig. 9.** The crystals produced on the bIm-modified membranes were very large and well intergrown with both size and intergrowth correlating positively to synthesis time. Images of the bIm-modified membranes are below in **Fig. 10.**

(a)



(b)



(c)

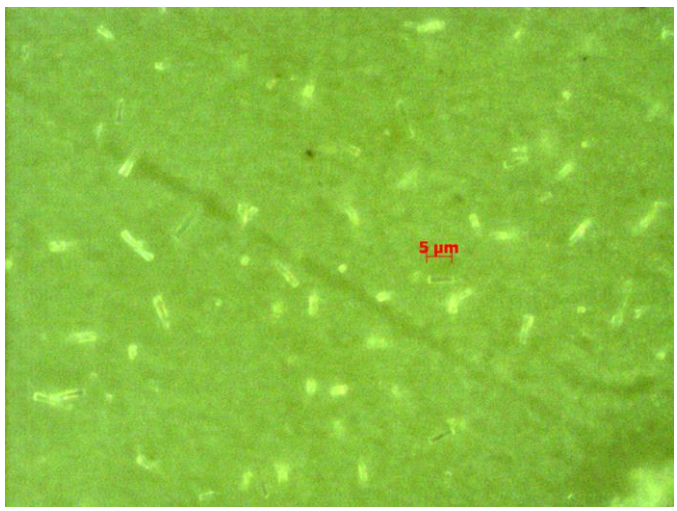


Fig. 9. Optical microscope images of mIm-modified membranes after (a) 8-hours growth time (b) 12-hours growth time and (c) 24-hours growth time. ZIF-11 crystals are not present.

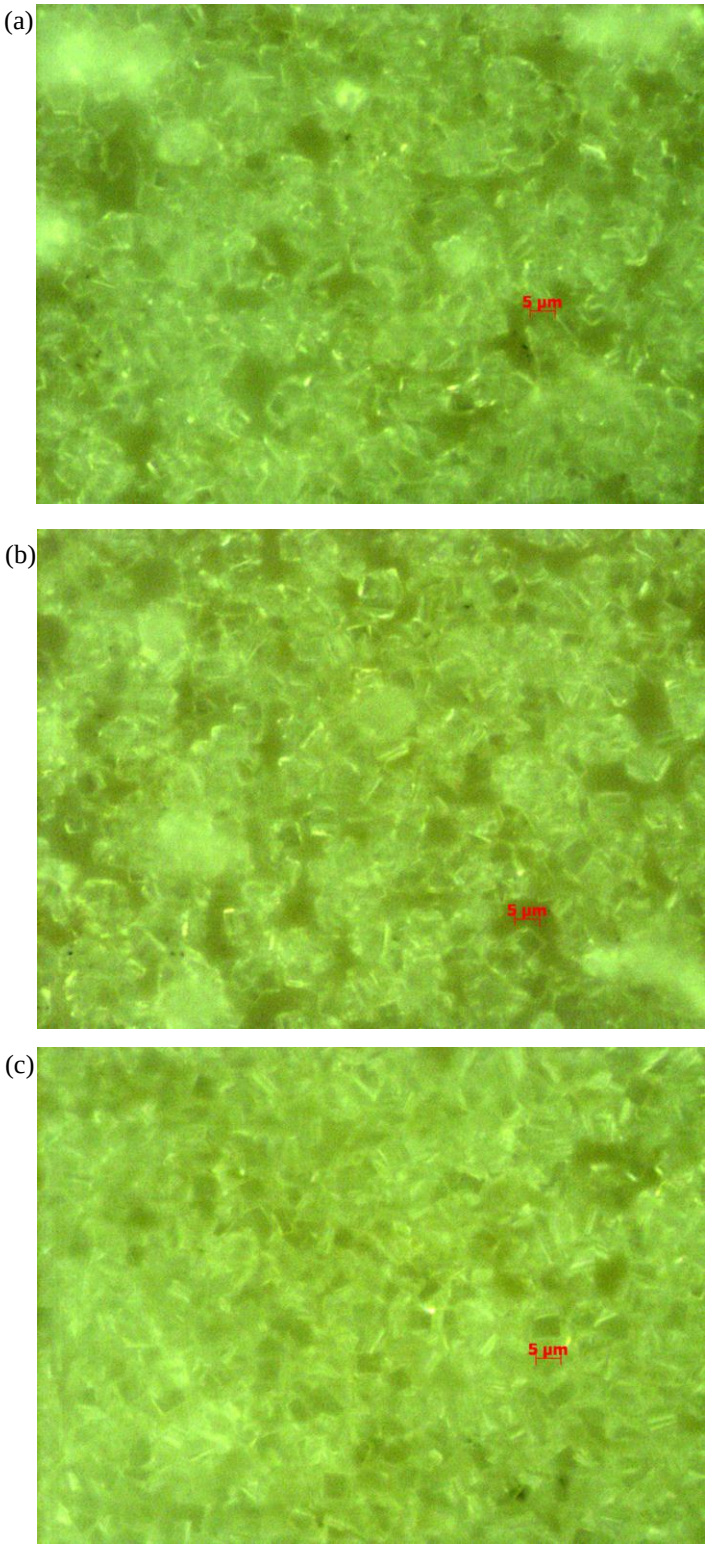
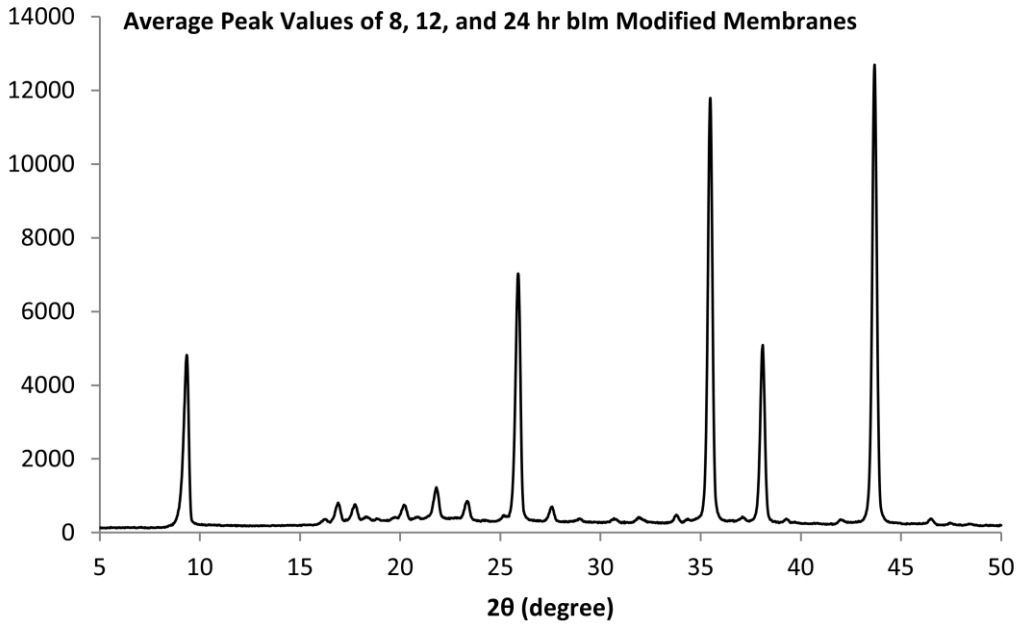


Fig. 10. Optical microscope images of bIm-modified membranes after (a) 8-hours growth time (b) 12-hours growth time and (c) 24-hours growth time. bIm-modified membranes exhibit large well-intergrown crystals. ZIF-11 crystals are not present.

(a)



(b)

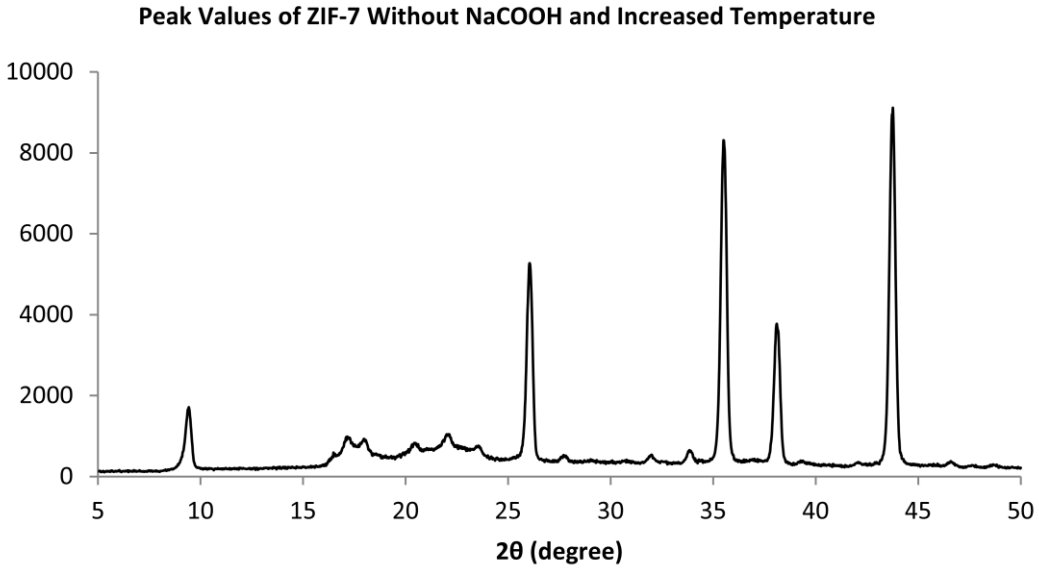


Fig. 11. X-Ray diffraction pattern of (a) the average peak values of the 8, 12, and 24 hour bIm-modified membranes and (b) The XRD pattern from a trial performed by the Jeong Group of ZIF-7 without NaCOOH and with a greatly increased temperature. ZIF-11 crystals are not present.

Table 2
Observed and peaks of unknown crystal structure obtained through attempted ZIF-11 and ZIF-7 synthesis.

bIm-modified Average Peaks	High Rate Unknown Structure
2θ	2θ
9.32	9.42
16.9	17.1
17.68	17.96
20.16	20.42
21.8	22
23.32	23.44
25.88	26.04
27.58	27.7
35.48	35.5
38.1	38.1
43.68	43.76

XRD measurements, shown above in **Fig. 11.**, were taken and neither the mIm nor the bIm-modified membranes matched the known ZIF-11 pattern. However, the pattern produced by the bIm-modified membranes was the same as a pattern that the Jeong Group encountered when the reaction rate of the synthesis of ZIF-7 was greatly increased due to temperature. The peaks line up closely as shown in **Table. 2** above.

CHAPTER IV

CONCLUSIONS

First attempt to synthesize ZIF-11 membranes

The proper structure (ZIF-11) was successfully synthesized in the first powder synthesis, but the following attempts to create well-intergrown ZIF-11 films proved difficult. The first obstacle was a lack of crystals on the surface of the alumina supports, resulting in too few crystals to get a conclusive XRD pattern. In attempts to increase the number and size of the crystals, the concentrations of the benzimidazole and zinc nitrate were doubled, resulting in formation of the incorrect crystal structure when XRD measurements were compared to the data in the literature. Only increasing the amount of zinc nitrate, benzimidazole, and sodium formate used for synthesis and keeping the amount of solvent (DEF) the same will not work. This could be due to the NaCOOH, acting as a base and deprotonator, increasing the pH too much to allow the growth of ZIF-11 crystals [22].

Increased synthesis time to produce ZIF-11 membranes

In order to produce more crystals that are larger, well-intergrown, and of the desired structure, the reaction time was increased from 4 hours to 8, 12, and 24 hrs, all using the original recipe. This produced membranes with large, uniform, well-intergrown crystals on the supports that were modified using a bIm in MeOH solution. The mIm-modified supports did not have many crystals as shown by the microscopic images, and the

crystals that were present were not of the same structural form as those observed on the bIm-modified membranes. The XRD pattern measurements of the mIm-modified membranes had no discernable peaks, and the bIm-modified membranes showed that the desired structure, ZIF-11, had not been formed. Furthermore, XRD measurements of the powder gathered from the autoclave apparatus (crystals that do not attach to the surface of the membrane support and sink to the bottom of the container) matched the pattern of the unknown structure on the bIm-modified membranes.

Surface modification with ligands

Although the incorrect crystal structure was formed on the supports, the surface modification process for ZIF membrane synthesis, as develop by the Jeong Research Group, is proving to be an effective method to produce large, well-intergrown crystals on the surface of the bIm-modified α-alumina supports for ZIF-11 membranes.

Unknown crystal structure

The XRD patterns showing that the crystals on the membranes and the powder from the same container were the same undesired and unknown structure suggest that the recipe used does not produce ZIF-11 consistently. It seems that the synthesis of ZIF-11 is very sensitive to the concentrations of the various chemical species, the amount of solvent, and the pH of the solution. The unknown pattern produced by the bIm-modified membranes was the same as a pattern that the Jeong Group encountered while working with ZIF-7, a ZIF that is made with the same ligands as ZIF-11. The reaction rate of the

synthesis of ZIF-7 was greatly increased due to temperature and the unknown crystal structure was produced. By reducing the amount of NaCOOH by 75%, ZIF-7 was correctly synthesized. In the same way, perhaps by decreasing the NaCOOH used to form the ZIF-11 crystals, the proper structure can be produced.

Future experiments and research

Further experiments should be done to perfect and fine-tune a synthesis process that consistently produces ZIF-11 crystals. After this recipe is found it will be possible to progress to forming ZIF-11 films and subsequently testing for hydrogen gas separation capabilities. ZIF membranes for the purpose of gas separation remain an exciting and promising technology for the future and ought to be explored further.

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